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# RELATIONSHIPS BETWEEN STRUCTURE AND RETENTION INDEX FOR N-SUBSTITUTED AMIDES OF ALIPHATIC ACIDS ON A NON-POLAR COLUMN

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## SUMMARY

The retention indices of 100 amides of aliphatic acids ($R^1$–CO–NH$R^2$) divided into ten series depending on the substituent $R^1$ at the carboxylic carbon atom or the substituent $R^2$ at the nitrogen atom, each containing the same set of ten substituents at the second site, have been measured on a non-polar GE SE-30 column. A relationship between the retention index of the amide and the number of primary, secondary, tertiary and quaternary carbon atoms in the molecule has been found. The retention indices obtained were correlated with those of corresponding simple model compounds, such as substituted hydrocarbons ($R^1$–H and $R^2$–H) and amines ($R^2$–$NH_2$). The good linear correlations obtained indicate that, for prediction of retention indices of amides the correlation method should be used instead of the additivity rule. Retention indices of amides were found to obey a two-parameter linear equation with the retention indices of corresponding model compounds as independent variables.

## INTRODUCTION

The possibility of fairly accurate prediction of retention indices plays a very important rôle in the synthesis and analysis of organic compounds.

There are several methods for the prediction of retention indices. Most of them consider the retention index as an additive function, and the simplest ones assume that the retention indices of compounds containing a certain functional group can be calculated by addition of a specific retention increment for this group to the retention indices of the corresponding hydrocarbons[1,2]. These methods, although not very precise, are commonly used because of their simplicity.

Recently Oszczapowicz *et al.*[3–8] have shown that retention indices of compounds, $I$(Cpd), correlate very well with the retention indices of the corresponding model compounds, $I$(Std), taken as standards:

$$I(\text{Cpd}) = a \cdot I(\text{Std}) + b \quad (1)$$

They have pointed out[3] that the additivity rule, used for prediction of retention indices

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TABLE I

RETENTION INDICES OF AMIDES ($R^1CONHR^2$) ON A GE SE-30 NON-POLAR COLUMN AT 180°C

| *No.* | $R^1$ | $R^2$ | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $CH_3$ | $C_2H_5$ | $n\text{-}C_3H_7$ | $iso\text{-}C_3H_7$ | $n\text{-}C_4H_9$ | $iso\text{-}C_4H_9$ | $tert.\text{-}C_4H_9$ | $n\text{-}C_6H_{13}$ | *Cyclohexyl* | *Phenyl* |
| 1 | H | 793 ± 6 | 794 ± 6 | 934 ± 4 | 911 ± 6 | 1067 ± 5 | 1014 ± 6 | 915 ± 3 | 1274 ± 2 | 1306 ± 4 | 1386 ± 8 |
| 2 | $CH_3$ | 857 ± 8 | 890 ± 5 | 974 ± 4 | 895 ± 3 | 1090 ± 3 | 1013 ± 2 | 900 ± 4 | 1292 ± 4 | 1313 ± 3 | 1429 ± 9 |
| 3 | $C_2H_5$ | 921 ± 4 | 950 ± 4 | 1040 ± 2 | 979 ± 4 | 1140 ± 4 | 1091 ± 3 | 969 ± 0 | 1353 ± 5 | 1378 ± 1 | 1480 ± 2 |
| 4 | $n\text{-}C_3H_7$ | 1005 ± 3 | 1044 ± 6 | 1127 ± 3 | 1042 ± 4 | 1228 ± 3 | 1167 ± 1 | 1048 ± 2 | 1434 ± 2 | 1459 ± 4 | 1531 ± 5 |
| 5 | $iso\text{-}C_3H_7$ | 940 ± 5 | 1002 ± 3 | 1090 ± 2 | 987 ± 2 | 1169 ± 4 | 1116 ± 3 | 983 ± 0 | 1352 ± 3 | 1392 ± 1 | 1484 ± 3 |
| 6 | Cyclopropyl | 1056 ± 5 | 1089 ± 6 | 1197 ± 5 | 1107 ± 3 | 1293 ± 4 | 1249 ± 2 | 1104 ± 4 | 1471 ± 3 | 1512 ± 4 | 1616 ± 6 |
| 7 | $n\text{-}C_4H_9$ | 1105 ± 3 | 1141 ± 5 | 1237 ± 4 | 1152 ± 5 | 1338 ± 3 | 1281 ± 1 | 1156 ± 3 | 1516 ± 4 | 1551 ± 5 | 1642 ± 3 |
| 8 | $iso\text{-}C_4H_9$ | 1069 ± 3 | 1103 ± 4 | 1165 ± 2 | 1088 ± 3 | 1261 ± 1 | 1218 ± 2 | 1101 ± 1 | 1469 ± 4 | 1496 ± 4 | 1571 ± 4 |
| 9 | $tert.\text{-}C_4H_9$ | 959 ± 6 | 1000 ± 5 | 1077 ± 2 | 1016 ± 4 | 1181 ± 1 | 1136 ± 2 | 1002 ± 2 | 1386 ± 5 | 1398 ± 3 | 1465 ± 3 |
| 10 | $n\text{-}C_5H_{11}$ | 1123 ± 2 | 1216 ± 2 | 1319 ± 3 | 1258 ± 3 | 1425 ± 3 | 1372 ± 1 | 1246 ± 1 | 1642 ± 4 | 1645 ± 3 | 1743 ± 4 |

of organic compounds, should be regarded as a very special case of linear regression, where the slope, $a$, of the correlation line is by definition equal to unity. It has been found that in many cases this coefficient is considerably different from unity and it was concluded that the use of the additivity rule for prediction of retention indices of some compounds may cause discernible errors and that the correlation method should be used instead.

It has been shown that for compounds containing two variable substituents the best results are obtained when dual parameter regression is used[4,5,7,8]:

$$I(\text{Cpd}) = a_1 \cdot I(\text{Std}_1) + a_2 \cdot I(\text{Std}_2) + b \qquad (2)$$

Thus the question arose as to how far the additivity rule can be applied for prediction of retention indices of monosubstituted amides and whether the methods presented above will provide better results in this case too.

In this work the Kováts retention indices[9,10] for 100 amides of aliphatic carboxylic acids ($R^1$–CO–NH$R^2$) have been measured on a non-polar GE SE-30 column. The compounds investigated can be divided into ten series depending on the substituent $R^1$ at the carboxylic carbon atom or the substituents $R^2$ at the nitrogen atom. Each series contained the same set of ten variable substituents at the second site. The substituents $R^1$ and $R^2$ are listed in Table I.

## EXPERIMENTAL

### *Materials*

All amides studied were synthesized in our laboratory by treating a primary amine with an equimolar amount of the corresponding acyl chloride. The amines were commercial samples from Merck or Fluka. Acyl chlorides were prepared by reaction of the corresponding carboxylic acids with oxalyl chloride. *n*-Alkanes $C_{10}$–$C_{15}$ were obtained from Applied Science Labs.

### *Gas chromatography*

A Chromatron Model GCHF 18.3.4 gas chromatograph with a flame ionization detector, connected on line with a microcomputer and equipped with a 1 m × 3 mm I.D. column packed with 15% GE SE-30 silicone gum rubber on Chromosorb W AW (60–80 mesh), was used. The column temperature was maintained at 180°C. The carrier gas was nitrogen at a flow-rate of 25 ml/min. Samples of 0.5 μl of solutions in pyridine or quinoline were injected by means of a 10-μl Hamilton syringe.

Retention indices and dead times were determined by regression analysis by the method of Grobler and Bálizs[11] as improved by Haken *et al.*[12] using the series of six $C_{10}$–$C_{15}$ *n*-alkanes, each time under the same conditions as used for the sample studied.

## RESULTS AND DISCUSSION

The retention indices obtained with confidence intervals at a significance level of 0.05, calculated from at least five measurements, are listed in Table I.

Searching for relationships between the retention indices and the structures of

the amides, our attention was drawn to the possibility of calculation of retention indices on the basis of the number of primary, secondary, tertiary and quaternary carbon atoms in the alkyl substituents $R^1$ and $R^2$ according to

$$I(\text{Cpd}) = \sum_{i=1}^{4} n^i I^i + I^0 \tag{3}$$

where $n^i$ = number of primary, secondary, tertiary and quaternary carbon atoms respectively, $I^i$ = increment of primary, secondary, tertiary and quaternary carbon atom respectively and $I^0$ = increment characteristic for monosubstituted amides. All calculations were made by means of the least-squares method. The confidence intervals were calculated at a significance level of 0.05.

The regression coefficients, $R$, obtained and Exner's $\Psi^\star$ function[13], as well as increments for carbon atoms calculated on the basis of eqn. 3, are presented in Table II.

The results indicated that for amides containing aliphatic substituents $R^1$ and $R^2$, the retention index depends on the number of carbon atoms, on whether these atoms are primary, secondary, tertiary or quaternary, and may also depend on the site of attachment to the functional group

$$I(\text{Cpd}) = \sum_{i=1}^{4} n^i(R^1) \cdot I^i(R^1) + \sum_{i=1}^{4} n^i(R^2) \cdot I^i(R^2) + I^0 \tag{4}$$

where $n^i(R^1), n^i(R^2)$ = number of primary, secondary, tertiary and quaternary carbon atoms in substituents $R^1$ and $R^2$, respectively and $I^i(R^1)$, $I^i(R^2)$ = increment of primary, secondary, tertiary and quaternary carbon atoms in substituents $R^1$ and $R^2$, respectively. The parameters obtained given in Table II (eight parameter scheme)

TABLE II

INCREMENTS FOR CALCULATION OF RETENTION INDICES

Without $R^1$–CO–$NHCH_3$ series. $n$ represents the number of retention indices considered.

| *Four parameters (eqn. 3)* | *Eight parameters (eqn. 4)* |
|---|---|
| $I^{I} = 38.8 \pm 6.6$ | $I^{I}(R^1) = 33.1 \pm 6.6$ |
| $I^{II} = 93.5 \pm 2.5$ | $I^{II}(R^1) = 88.0 \pm 2.5$ |
| $I^{III} = 79.5 \pm 6.8$ | $I^{III}(R^1) = 78.1 \pm 8.2$ |
| $I^{IV} = 35.9 \pm 14.4$ | $I^{IV}(R^1) = 51.5 \pm 17.1$ |
| – | $I^{I}(R^2) = 52.1 \pm 9.4$ |
| – | $I^{II}(R^2) = 98.7 \pm 3.2$ |
| – | $I^{III}(R^2) = 76.5 \pm 7.7$ |
| – | $I^{IV}(R^2) = 9.9 \pm 17.2$ |
| $I^0 = 698.1$ | $I^0 = 687.4$ |
| $R = 0.9956$ | $R = 0.9972$ |
| $\Psi = 0.0973$ | $\Psi = 0.0799$ |
| $n = 72$ | $n = 72$ |

* $\Psi = [n(1 - r^2)/f]^{1/2}$, where $r$ is the correlation coefficient, $n$ the number of experimental points and $f$ the number of degrees of freedom.

shows that indeed for primary and quaternary carbon atoms the increment depends on the site of attachment whereas for secondary and tertiary atoms it seems independent.

Searching for the best methods of prediction of retention indices of amides with substituents of any type, we have calculated the linear regression between the retention indices of amides of each series and those of appropriate model compounds. For each series containing a constant substituent $R^2$ at the nitrogen atom, we have correlated the retention indices of amides with those of corresponding hydrocarbons ($R^1$–H or $R^1$–$CH_3$). The regression coefficients, *a* and *b* (eqn. 1), and the estimators of correlation, *i.e.*, the correlation coefficient, *r*, and Exner's $\Psi$ function are given in Table III. For each series the slope, *a*, of the regression lines is markedly different from unity and application of the additivity rule, instead of this method, may result in considerable errors. The regressions obtained indicate that, for all the series studied, the correlations with the retention indices of hydrocarbons $R^1$–H are very good, whereas the correlations with the retention indices of hydrocarbons $R^1$–$CH_3$ are very good for the first three series but for the other series are only satisfactory. The results presented show that, as model compounds for the prediction of retention indices of amides, hydrocarbons $R^1$–H should be used.

For series with an invariant substituent $R^1$ at the carboxylic group, we have

TABLE III

REGRESSION PARAMETERS OF RETENTION INDICES OF AMIDES ($R^1CONHR^2$) CONTAINING A CONSTANT SUBSTITUENT, $R^2$, AT THE NITROGEN ATOM *vs.* RETENTION INDICES OF CORRESPONDING HYDROCARBONS A ($R^1H$) AND B ($R^1CH_3$) (EQN. 1)

| *No.* | $R^2$ | *Type* | *a* | *b* | *r* | $\Psi$ | *n* |
|---|---|---|---|---|---|---|---|
| 1 | $CH_3$ | A | 0.716 ± 0.231 | 787.4 | 0.9849 | 0.2232 | 5 |
| | | B | 0.708 ± 0.132 | 719.5 | 0.9910 | 0.1636 | 6 |
| 2 | $C_2H_5$ | A | 0.843 ± 0.115 | 795.3 | 0.9973 | 0.0956 | 5 |
| | | B | 0.845 ± 0.066 | 710.1 | 0.9984 | 0.0686 | 6 |
| 3 | *n*-$C_3H_7$ | A | 0.887 ± 0.129 | 873.3 | 0.9969 | 0.1022 | 5 |
| | | B | 0.800 ± 0.157 | 825.1 | 0.9901 | 0.1718 | 6 |
| 4 | iso-$C_3H_7$ | A | 0.899 ± 0.167 | 795.5 | 0.9949 | 0.1296 | 5 |
| | | B | 0.734 ± 0.281 | 782.6 | 0.9640 | 0.3257 | 6 |
| 5 | *n*-$C_4H_9$ | A | 0.868 ± 0.188 | 983.8 | 0.9932 | 0.1508 | 5 |
| | | B | 0.749 ± 0.219 | 952.5 | 0.9786 | 0.2519 | 6 |
| 6 | iso-$C_4H_9$ | A | 0.908 ± 0.128 | 912.4 | 0.9971 | 0.0985 | 5 |
| | | B | 0.763 ± 0.244 | 899.3 | 0.9745 | 0.2748 | 6 |
| 7 | *tert.*-$C_4H_9$ | A | 0.879 ± 0.130 | 800.1 | 0.9968 | 0.1036 | 5 |
| | | B | 0.715 ± 0.273 | 788.8 | 0.9641 | 0.3253 | 6 |
| 8 | *n*-$C_6H_{13}$ | A | 0.863 ± 0.213 | 1188.5 | 0.9911 | 0.1715 | 5 |
| | | B | 0.741 ± 0.230 | 1159.2 | 0.9758 | 0.2677 | 6 |
| 9 | Cyclohexyl | A | 0.837 ± 0.110 | 1218.1 | 0.9974 | 0.0923 | 5 |
| | | B | 0.711 ± 0.211 | 1193.0 | 0.9780 | 0.2558 | 6 |
| 10 | Phenyl | A | 0.790 ± 0.265 | 1328.0 | 0.9837 | 0.2319 | 5 |
| | | B | 0.707 ± 0.201 | 1287.7 | 0.9796 | 0.2461 | 6 |

TABLE IV

REGRESSION PARAMETERS OF RETENTION INDICES OF AMIDES ($R^1CONHR^2$) CONTAINING A CONSTANT SUBSTITUENT, $R^1$, AT THE CARBOXYLIC GROUP *vs.* RETENTION INDICES OF CORRESPONDING HYDROCARBONS ($R^2H$) (EQN. 1)

| *No.* | $R^1$ | *a* | *b* | *r* | $\Psi$ | *n* |
|---|---|---|---|---|---|---|
| 1 | H | 1.057 ± 0.175 | 635.4 | 0.9898 | 0.1688 | 7 |
| 2 | $CH_3$ | 0.962 ± 0.181 | 716.9 | 0.9868 | 0.1916 | 7 |
| 3 | $C_2H_5$ | 0.953 ± 0.170 | 780.1 | 0.9881 | 0.1817 | 7 |
| 4 | $n$-$C_3H_7$ | 0.914 ± 0.125 | 877.2 | 0.9930 | 0.1400 | 7 |
| 5 | iso-$C_3H_7$ | 0.892 ± 0.125 | 829.8 | 0.9927 | 0.1431 | 7 |
| 6 | Cyclopropyl | 0.932 ± 0.157 | 928.0 | 0.9895 | 0.1712 | 7 |
| 7 | $n$-$C_4H_9$ | 0.905 ± 0.136 | 981.4 | 0.9915 | 0.1537 | 7 |
| 8 | iso-$C_4H_9$ | 0.874 ± 0.153 | 938.0 | 0.9886 | 0.1780 | 7 |
| 9 | *tert.*-$C_4H_9$ | 0.886 ± 0.119 | 837.4 | 0.9932 | 0.1377 | 7 |
| 10 | $n$-$C_5H_{11}$ | 1.022 ± 0.112 | 1015.7 | 0.9955 | 0.1124 | 7 |

correlated the retention indices of amides with those of corresponding hydrocarbons ($R^2$–H) or primary amines[3] ($R^2$–$NH_2$), to determine whether the slopes are different from unity for both correlations and which of the standards tested ensure higher accuracy of prediction. The results obtained are presented in Tables IV and V, respectively. The regressions indicate that for all series studied the correlations with the retention indices of primary amines are of the highest quality and the correlation with the retention indices of hydrocarbons are still good.

It should be mentioned that we have found very good correlations of the retention indices of amides, containing a constant substituent $R^1$ at the carboxylic group, with those of the corresponding acetamides (Table VI).

The regression coefficients, *a*, for the series of amides studied containing a constant substituent $R^1$ at the carboxylic group were identical within the confidence intervals. The same was observed for the series of amides with a constant substituent

TABLE V

REGRESSION PARAMETERS OF RETENTION INDICES OF AMIDES ($R^1CONHR^2$) CONTAINING A CONSTANT SUBSTITUENT, $R^1$, AT THE CARBOXYLIC GROUP *vs.* RETENTION INDICES OF CORRESPONDING PRIMARY AMINES ($R^2NH_2$)* (EQN. 1)

| *No.* | $R^1$ | *a* | *b* | *r* | $\Psi$ | *n* |
|---|---|---|---|---|---|---|
| 1 | H | 0.966 ± 0.089 | 448.5 | 0.9957 | 0.1066 | 8 |
| 2 | $CH_3$ | 1.028 ± 0.087 | 419.0 | 0.9964 | 0.0973 | 8 |
| 3 | $C_2H_5$ | 0.991 ± 0.072 | 509.6 | 0.9974 | 0.0840 | 8 |
| 4 | $n$-$C_3H_7$ | 0.968 ± 0.115 | 600.9 | 0.9931 | 0.1358 | 8 |
| 5 | iso-$C_3H_7$ | 0.949 ± 0.119 | 555.9 | 0.9922 | 0.1438 | 8 |
| 6 | Cyclopropyl | 0.965 ± 0.100 | 667.3 | 0.9947 | 0.1187 | 8 |
| 7 | $n$-$C_4H_9$ | 0.940 ± 0.095 | 724.3 | 0.9949 | 0.1165 | 8 |
| 8 | iso-$C_4H_9$ | 0.949 ± 0.091 | 655.6 | 0.9954 | 0.1103 | 8 |
| 9 | *tert.*-$C_4H_9$ | 0.917 ± 0.112 | 588.4 | 0.9926 | 0.1402 | 8 |
| 10 | $n$-$C_5H_{11}$ | 0.971 ± 0.086 | 800.8 | 0.9961 | 0.1025 | 8 |

* According to ref. 3; the retention index of *tert.*-butylamine is 483 ± 6.

TABLE VI

REGRESSION PARAMETERS OF RETENTION INDICES OF AMIDES ($R^1CONHR^2$) CONTAINING A CONSTANT SUBSTITUENT, $R^1$, AT THE CARBOXYLIC GROUP *vs.* RETENTION INDICES OF CORRESPONDING ACETAMIDES ($CH_3CONHR^2$) (Eqn. 1)

| *No.* | $R^1$ | *a* | *b* | *r* | $\Psi$ | *n* |
|---|---|---|---|---|---|---|
| 1 | H | 1.017 ± 0.139 | −43.9 | 0.9861 | 0.1855 | 10 |
| 2 | $C_2H_5$ | 0.974 ± 0.036 | 92.2 | 0.9990 | 0.0506 | 10 |
| 3 | *n*-$C_3H_7$ | 0.947 ± 0.042 | 199.6 | 0.9985 | 0.0611 | 10 |
| 4 | iso-$C_3H_7$ | 0.930 ± 0.055 | 160.9 | 0.9974 | 0.0806 | 10 |
| 5 | Cyclopropyl | 0.959 ± 0.055 | 248.0 | 0.9975 | 0.0789 | 10 |
| 6 | *n*-$C_4H_9$ | 0.932 ± 0.038 | 319.4 | 0.9987 | 0.0563 | 10 |
| 7 | iso-$C_4H_9$ | 0.911 ± 0.045 | 283.5 | 0.9982 | 0.0675 | 10 |
| 8 | *tert.*-$C_4H_9$ | 0.907 ± 0.059 | 196.1 | 0.9968 | 0.0893 | 10 |
| 9 | *n*-$C_5H_{11}$ | 1.006 ± 0.110 | 327.5 | 0.9911 | 0.1488 | 10 |

$R^2$ at the nitrogen atom. This leads to the conclusion that, for the prediction of their retention indices, a two-parameter linear regression (eqn. 2) may be used. As a first standard series, for substituents at the nitrogen atom, we have chosen hydrocarbons $R^2$–H. As the second one (for substituents at the carboxylic carbon atom) we have used hydrocarbons $R^1$–H. Because the linear correlations with hydrocarbons $R^2$–H (Table IV) were of good quality, but not as good as for primary amines $R^2$–$NH_2$ (Table V), we have also calculated dual-parameter correlations using primary amines $R^2$–$NH_2$ and hydrocarbons $R^1$–H as standards. The results obtained are collected in Table VII. The correlations with both types of standards are of very good quality as indicated by both the correlation coefficient, *R*, and Exner's $\Psi$ function.

## CONCLUSION

We have compared the retention indices calculated on the basis of eqns. 2, 3 and 4 with the experimental values. The distribution of errors (Table VIII) indicates that, in the prediction of retention indices of aliphatic acid amides containing alkyl substituents at the nitrogen atom, the best results are obtained on the basis of eqn. 4. However, for amides containing other type of substituents, dual-parameter regression (eqn. 2) should be used.

TABLE VII

MULTIPLE REGRESSION PARAMETERS OF RETENTION INDICES OF AMIDES *vs.* RETENTION INDICES OF STANDARDS (EQN. 2)

| *Standards* | $a_1$ | $a_2$ | *b* | *R* | $\Psi$ | *n* |
|---|---|---|---|---|---|---|
| $R^1$H $R^2$H | 0.829 ± 0.078 | 0.951 ± 0.051 | 625.5 | 0.9917 | 0.1346 | 35 |
| $R^1$H $R^2NH_2$ | 0.866 ± 0.050 | 0.980 ± 0.038 | 351.0 | 0.9953 | 0.1011 | 40 |

TABLE VIII

DISTRIBUTION (%) OF ERRORS OF PREDICTIONS OF RETENTION INDICES BASED ON EQNS. 2, 3 AND 4

| *I(calc.) − I(exptl.)* | *Equation* | | | |
|---|---|---|---|---|
| | *2** | *2*** | *3* | *4* |
| 0–10 | 25.7 | 30.0 | 41.7 | 52.8 |
| 10–20 | 17.1 | 27.5 | 34.7 | 33.3 |
| 20–30 | 11.4 | 27.5 | 16.7 | 11.1 |
| 30–40 | 28.6 | 12.5 | 5.6 | 2.8 |
| 40–50 | 5.7 | – | 1.3 | – |
| 50–60 | 8.6 | 2.5 | – | – |
| 60–70 | – | – | – | – |
| 70–80 | 2.9 | – | – | – |
| Mean accuracy of prediction*** | 25 ± 6 | 18 ± 4 | 14 ± 3 | 11 ± 2 |

* $R^1H$ and $R^2H$ as the standards.
** $R^1H$ and $R^2NH_2$ as the standards.
*** In retention index units.

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